PATENT ABSTRACTS OF JAPAN

(11)Publication number : 09-268271

(43)Date of publication of application : 14.10.1997

(51)Int.Cl. C09D183/00
A61K 7/02
C07F 7/08
C08K 9/06

(21)Application number : 08-104417

(22)Date of filing : 30.03.1996

(71)Applicant : SHISEIDO CO LTD

(72)Inventor : FUKUI HIROSHI
KANAMARU TETSUYA
OSHIMA RIKA
KAWAURA TAKESHI

(54) PRODUCTION OF SILICONE-COATED POWDER

(57)Abstract:

PROBLEM TO BE SOLVED: To uniformly and stably obtain the subject powder high in water repellency, excellent in dispersibility in oils, and useful for coating materials, cosmetics, etc., safely for the environment at a low cost by bringing powder material into contact with a mixture solution comprising a silicone compound and an aqueous solution.

SOLUTION: This method for producing a silicone-coated powder comprises bringing (A) the powder of an organic pigment, an inorganic pigment, a metal (hydr)oxide, mica or a pearl-lustrous material into contact with (B) a mixture solution comprising a silicone compound [e.g. a silicone compound having one or more Si-H groups, concretely a silicone compound of the formula: (R1HSiO)a(R2R3SiO)b(R4R5R6SiO1/2)c[R1-R6 are each H, a 1-10C hydrocarbon group which may be substituted with a halogen atom, wherein R1-R3 are simultaneously not be H; (a) is ≥1; (b) is ≥2; (c) is 0, 2; 3≤ (a)+(b)+(c) ≤1000; the compound contains one or more Si-H groups], more concretely methylhydrogenpolysiloxane} and an aqueous solution, and, if necessary, subsequently heating the contact product.

LEGAL STATUS

[Date of request for examination] 24.11.2000

[Date of sending the examiner's decision of 30.07.2002

rejection]	
[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]	
[Date of final disposal for application]	
[Patent number]	3552843
[Date of registration]	14.05.2004
[Number of appeal against examiner's decision of rejection]	2002-16534
[Date of requesting appeal against examiner's decision of rejection]	29.08.2002
[Date of extinction of right]	

* NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

CLAIMS

[Claim(s)]
[Claim 1] The manufacture approach of the silicone covering fine particles characterized by contacting fine particles to the mixed solution which comes to mix a silicone compound in a drainage system solution.
[Claim 2] The manufacture approach of the silicone covering fine particles according to claim 1 characterized by heating after contacting fine particles to a mixed solution.
[Claim 3] The manufacture approach of silicone covering fine particles according to claim 1 or 2 that the above-mentioned silicone compound is a silicone compound which has a Si-H radical.
[Claim 4] The silicone compound which has the above-mentioned Si-H radical is the following general formula (I).
[Formula 1]
$(R^1HSiO)_a(R^2R^3SiO)_b(R^4R^5R^6SiO_{1/2})_c$ (I)

The inside of [type, R1, R2, and R3 are hydrogen atoms mutually-independent, or it is the hydrocarbon group of the replaceable carbon numbers 1-10 by at least one halogen atom. ; R4, (However, R1, R2, and R3 are not hydrogen atoms at coincidence) R5 And R6 is a hydrogen atom mutually-independent, or it is the hydrocarbon group of the replaceable carbon numbers 1-10 by at least one halogen atom, and;a is one or more integers. b -- zero -- or -- one -- more than -- an integer -- it is -- c -- zero -- or -- two -- it is (however, it is 3 <=a+b+c<=10000) --; -- and -- this -- a compound -- Si-H -- a base -- a part -- at least -- one -- a piece -- containing -- a thing -- ** -- carrying out --] -- expressing -- having -- silicone -- a compound -- it is -- being according to claim 3 -- silicone -- covering -- fine particles -- manufacture -- an approach .
[Claim 5] The manufacture approach of silicone covering fine particles according to claim 4 that the silicone compound which has the above-mentioned Si-H radical is methil hydrogen polysiloxane.
[Claim 6] The manufacture approach of silicone covering fine particles given in any 1 term of claims 1-5 whose above-mentioned fine particles are one sort or two sorts or more of combination of an organic pigment, an inorganic pigment, a metallic oxide, a metal hydroxide, a mica, pearl gloss material, a metal, magnetic fine particles, a silicate mineral, or the porous matter.
[Claim 7] The manufacture approach of silicone covering fine particles given in any 1 term of claims 1-6 whose mixed rates of the above-mentioned drainage system solution and a silicone compound are the drainage system solutions 0.1-500 (weight ratio) to the silicone compound 1.
[Claim 8] The manufacture approach of silicone covering fine particles given in any 1 term of claims 1-6 whose mixed rates of the above-mentioned drainage system solution and a silicone compound are the drainage system solutions 1-200 (weight ratio) to the silicone compound 1.
[Claim 9] The manufacture approach of silicone covering fine particles given in any 1 term of claims 1-8 whose amount of silicone compounds to the above-mentioned fine particles is 0.1 - 20 % of the weight.
[Claim 10] The manufacture approach of silicone covering fine particles given in any 1 term of claims

1-8 whose amount of silicone compounds to the above-mentioned fine particles is 0.5 - 15 % of the weight.
[Claim 11] The manufacture approach of silicone covering fine particles given in any 1 term of claims 1-10 which adds any one or more sorts in alkali metal and alkaline earth metal to the above-mentioned mixed solution, and adds an acid further after crosslinking reaction.
[Claim 12] Reforming processing fine particles which come to add the compound which has a pendant radical further to the Si-H radical of the silicone covering fine particles manufactured by the approach according to claim 3 to 11.

[Translation done.]

* NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention is the environment top insurance which starts the manufacture approach of silicone covering fine particles, and covers a fine-particles front face with a silicone compound to homogeneity and stability in more detail using a drainage system solvent, without using an organic solvent, and relates to the manufacture approach of silicone covering fine particles that the manufacturing cost was reduction-ized.
[0002] this invention -- the manufactured silicone covering fine particles have high water repellence, and it often distributes them in fats and oils. Therefore, the manufacture approach of this invention covers a pigment with a silicone compound, and since a vivid color comes out when this is blended with an oil wax system, it is widely applicable to fields, such as a coating, ink, cosmetics, and a medical ingredient.
[0003]
[Description of the Prior Art] As the manufacture approach of silicone covering fine particles, conventionally, after covering a fine-particles front face with a silicone compound, the technique of making covering of a up to [fine particles] stable is proposed by the approach of performing or processing baking by a sodium hydroxide etc. And the silicone covering fine particles obtained by doing in this way add the compounds (for example, unsaturated compound etc.) which have a desired functional group (pendant radical) further, and are used as reforming fine particles which gave the specific property to fine particles.
[0004] However, even if baking, a sodium hydroxide, etc. perform stabilizing treatment, it is important that the silicone compound is covered by homogeneity as the premise at fine particles. it is difficult to come out comparatively, to use a silicone compound less than 5% of the weight of often to fine particles, generally, and to cover a silicone compound with an amount smaller than this to homogeneity at fine particles. Therefore, after making it dissolve in a solvent, seeing a silicone compound, making the upper amount increase and making fine particles cover the silicone compound using this to homogeneity to some extent, generally performing baking is performed. However, the problem of removal of the solvent in which silicone is dissolved in this case arises. That is, as a solvent in which a silicone compound is dissolved, although organic solvents, such as a hexane, toluene, a xylene, and chloroform, are used, for an environment, neither is desirable and also generates the problem of ignition at the time of heating. Though it processes by the closed system in order to prevent this, it is difficult to prevent leakage completely, and it also has the problem of the amount of organic solvent remaining in fine particles.
[0005] The method of on the other hand performing silicone covering by the drainage system, without using an organic solvent is indicated by JP,5-32914,A etc. This tends to make it perform silicone covering by contacting to fine particles what made the silicone particle detailed, and water was made to distribute this, and was made into the silicone emulsion. In order to make the emulsion of a certain thing form, it is necessary to use a surfactant, therefore this surfactant sticks to a silicone compound, and

remains, and the big problem of spoiling special water repellence has the advantage that this approach can contact a silicone compound to fine particles at homogeneity. Therefore, although the need has the activity of removing a surfactant by filtration when it manufactures by this approach, the problem mentioned above when it could not remove easily with water and the organic solvent washed will produce the surfactant which stuck to the silicone compound.

[0006] Therefore, the present condition is that development of the approach of covering fine particles with a silicone compound to homogeneity and stability is desired, without using on an environment the organic solvent which is not desirable, the difficult surfactant of removal, etc.

[0007]

[Problem(s) to be Solved by the Invention] This invention was made in view of the above-mentioned situation, and the technical problem is in simple and providing insurance with the manufacture approach of the silicone covering fine particles which can moreover cover silicone to stability on fine particles at homogeneity.

[0008]

[Means for Solving the Problem] It found out that silicone covering uniform and stabilized on the fine-particles front face could be performed by this invention persons' contacting to fine particles what was made to mix a silicone compound and a drainage system solution, and was made into mixed liquor as a result of repeating research wholeheartedly, and heating by request further.

[0009] this invention persons in more detail a silicone compound / drainage system solution Although it is not necessarily uniform in the state of separation when making fine particles contact, silicon oil spreads on a drainage system solution front face thinly, and forms the silicone film in it. When this film contacted fine particles and moisture was removed by forming the thin film on fine particles and heating by request further, based on a header and this, it came to complete this invention for silicone being covered by fine particles at homogeneity. Moreover, when using the silicone compound which has especially a Si-H radical, water is indispensable to crosslinking reaction and it is also effective in silicone film formation that this exists. As far as this invention persons get to know, the example of processing of a silicone compound / drainage system solution which examined the system which is not dissolved mutually is not made at all other than the conventional example which used the above-mentioned silicone emulsion.

[0010] That is, this invention offers the manufacture approach of the silicone covering fine particles characterized by contacting fine particles to the mixed solution which comes to mix a silicone compound in a drainage system solution. Here, it is desirable to heat fine particles after contacting to the above-mentioned mixed solution.

[0011] Moreover, this invention offers the reforming processing fine particles which come to add the compound which has a pendant radical further to the Si-H radical of the silicone covering fine particles manufactured by the above-mentioned approach.

[0012] Since the silicone covering fine particles obtained by this invention are stable, do not have an interaction with drugs and do not have disintegration to perfume further, when it uses for drugs, cosmetics, etc., its stability with the passage of time improves remarkably. Moreover, since the addition consistency to a Si-H radical and the fine particles of the compound (Si-H radical reactivity compound) which can react is high and control of dispersibility can be performed, the magnetic material which was excellent by processing magnetic fine particles by this invention approach can be obtained. Since the higher theoretical plate number can be sharply made high for separation of the bulking agent for column chromatographs by making an addition consistency high, it can use for the column packing material for gas chromatographs, the column packing material for liquid chromatographs, etc. further again.

[0013]

[Embodiment of the Invention] Hereafter, this invention is explained in full detail.

[0014] If the silicone compound used by this invention has low viscosity near the boiling point of water even if it is not limited especially if it is a silicone system, and it can use silicon oil, silicone resin, silicone wax, etc., for example, viscosity differs (preferably 100cps or less extent), silicone covering processing can be performed more to fitness.

[0015] Also in these, the silicone compound which has a Si-H radical is used especially suitably. This is because the direction at the time of Si-H radicals constructing a bridge and forming the network structure on a fine-particles front face cannot **** easily. Moreover, since hydrogen may occur at the time of covering processing when the silicone compound which has a Si-H radical is used, especially the processing by the drainage system is effective. In addition, when carrying out covering processing with the silicone compound which has a Si-H radical, it may become the cause of hydrogen generating at the time of manufacture of the product with which survival of the Si-H radical after a reaction contained silicone covering fine particles. Since water lives together by the manufacture approach of this invention, it is safe, even if a Si-H radical and water react and hydrogen occurs.
[0016] In addition, even if Si-OH radical exists as functional groups other than a Si-H radical, the same effectiveness as the above is expectable. What can give functionality to fine particles is mentioned as the other functional group. Specifically, in hydrophobicity, a polyoxyethylene, or a glycerol radical, a hydrophilic property and a fluoro alkyl group can give [an alkyl group or a phenyl group] a canal and non-dense oiliness, respectively. In addition, an epoxy group, the amino group, a carboxyl group, quarternary ammonium salt, etc. are mentioned by the property. Two or more sorts of these radicals may exist in 1 molecule.
[0017] As a silicone compound which has a Si-H radical, it is the following general formula (I), for example.
[0018]
[Formula 2]

$$(R^1HSiO)_a(R^2R^3SiO)_b(R^4R^5R^6SiO_{1/2})_c \quad (I)$$

The inside of [type, R1, R2, and R3 are hydrogen atoms mutually-independent, or it is the hydrocarbon group of the replaceable carbon numbers 1-10 by at least one halogen atom. ; R4, (However, R1, R2, and R3 are not hydrogen atoms at coincidence) R5 And R6 is a hydrogen atom mutually-independent, or it is the hydrocarbon group of the replaceable carbon numbers 1-10 by at least one halogen atom, and;a is one or more integers. b -- zero -- or -- one -- more than -- an integer -- it is -- c -- zero -- or -- two -- it is (however, it is 3 <=a+b+c<=10000) --; -- and -- this -- a compound -- Si-H -- a base -- a part -- at least -- one -- a piece -- containing -- a thing -- ** -- carrying out --] -- expressing -- having -- silicone -- a compound -- suitable -- using -- having .
[0019] In the case of c= 0, it is the following general formula (II) here.
[0020]
[Formula 3]

$$(R^1HSiO)_a(R^2R^3SiO)_b \quad (II)$$

As having defined R1, R2, R3, a, and b above among [type. However, it is R1 and R2 preferably. And R3 It is the low-grade alkyl group or aryl group (for example, phenyl group) of the replaceable carbon numbers 1-4 by at least one halogen atom (especially fluorine atom) mutually-independent,;a+b is three or more, and they are 10-1000, and the annular silicone compound especially expressed with] which is 20-500 preferably. That to which two or more hydrogen atoms exist in 1 molecule preferably is desirable.
[0021] Moreover, in the case of c= 2, it is the following general formula (III).
[0022]
[Formula 4]

$$(R^1HSiO)_a(R^2R^3SiO)_b(R^4R^5R^6SiO_{1/2}) \quad (III)$$

As having defined R1 -R6, a, and b above among [type. However, it is R1 -R6 preferably. It is the low-grade alkyl group or aryl group (for example, phenyl group) of the replaceable carbon numbers 1-4 by at least one halogen atom (especially fluorine atom) mutually-independent, and;a+b is 10-1000, and the chain-like silicone compound by which it is especially expressed with] which is 20-50. Methil hydrogen polysiloxane etc. can be mentioned as an example of a general formula (III).

[0023] The drainage system solution used by this invention is a solution which uses water as a principal component, and means the solution which water contains about 80% of the weight or more. As other components other than water, alcohols, such as ethanol, a methanol, and isopropanol, a sodium hydroxide, a potassium hydroxide, a calcium hydroxide, a hydrochloric acid, sulfuric acids, such mixture, etc. are mentioned. In addition, as water contained in this drainage system solution, although ion exchange water, distilled water, etc. are mentioned, inorganic ion may exist in this water. As inorganic ion, Li+, Na+, K+, Rb+, Cs+, Mg++, calcium++, HSO4-, Cl-, etc. are mentioned. With these ion, bridge formation of a Si-H radical can be promoted under alkali. In addition, the organic acid and the organic amine may be added.

[0024] Although a silicone compound is mixed with the above-mentioned drainage system solution and considered as a mixed solution, it is desirable the drainage system solutions 0.1-500 and to mix both mixed rate in 1-200 (weight ratio) more suitably to the silicone compound 1. A mixed approach can be performed using the mixer which can carry out with a conventional method, for example, is usually used. When carrying out wet blending, the rate of a drainage system solution is made [many], and in the case of dry blending, lessening the rate of a drainage system solution etc. can adjust suitably.

[0025] Next, fine particles are contacted to the above-mentioned mixed solution, and it heats by the case.

[0026] Although especially the fine particles used for this invention are not restricted, generally the body (a larger thing than 10mm may also be contained) of arbitration with a particle size of 10mm or less is meant, and, specifically, an organic pigment, an inorganic pigment, a metallic oxide and a metal hydroxide, a mica, a pearl gloss ingredient, a metal, carbon, magnetic powder, a silicate mineral, a porous material, etc. are mentioned in instantiation. At least one kind of these fine particles may be used again combining plurality, and may be floc, a Plastic solid, or a molding object. moreover, a fine-particles top -- or other matter (for example, a coloring agent, UV absorbent, drugs, various additives) may be contained in it.

[0027] As an organic pigment, for example Red No. 201, red No. 202, red No. 204, Red No. 205, red No. 220, red No. 226, red No. 228, red No. 305, Orange No. 203, orange No. 204, yellow No. 205, yellow No. 401, and blue No. 404, Furthermore, red No. 3, red No. 104, red No. 106, red No. 227, red No. 230, Red No. 401, red No. 505, orange No. 205, yellow No. 4, yellow No. 5, yellow No. 202, yellow No. 203, green No. 3, blue No. 1, etc. may be mentioned, and things, such as a zirconium lake, a barium lake, or an aluminium lake, are further sufficient as these organic pigments.

[0028] As an inorganic pigment, Berlin blue, ultramarine blue, manganese violet, bismuth oxychloride, etc. are mentioned, for example.

[0029] As a metallic oxide and a metal hydroxide, for example A magnesium oxide, A magnesium hydroxide, a calcium oxide, a calcium hydroxide, an aluminum oxide, an aluminum hydroxide, a silica, and an iron oxide (alpha-Fe 2O3 and gamma-Fe 2O3 --) Synthetic Ochre (especially rod-like thing), such as Fe 3O4 and FeO, a red iron oxide, Black iron oxide, an iron hydroxide, titanium oxide (especially titanium dioxide with a particle size of 0.001-0.1 micrometers), Low hypo---ic acid-ized titanium, a zirconium dioxide, chrome oxide, chromium hydroxide, manganese oxide, Cobalt oxide, nickel oxide, the multiple oxide by two or more sorts of such combination and a compound hydroxide, for example, a silica alumina, titanic-acid iron, titanic-acid cobalt, lithium cobalt titanate, ulmin acid cobalt, etc. are mentioned.

[0030] As a mica, it is a muscovite, phlogopite, a biotite, sericite, an iron mica, lepidolite, a lithia mica, a CHINWARUDO mica, a paragonite, synthetic mica or KAl2 O(aluminum and Si3)10F2, KMg3 O (aluminum and Si3)10F2, and K(Mg and Fe3) (aluminum and Si3) O10F2, for example. The mica expressed is mentioned.

[0031] As a pearl gloss ingredient, mica titanium system composite material, mica ferrous-oxide system composite material, bismuth oxy-chloride, a guanine, the mica (for example, a titanium oxide covering mica, a ferrous-oxide covering mica) further covered with the titanium compound containing oxidization titanium nitride and/or low hypo---ic acid-ized titanium are mentioned, for example. About the titanium of mica titanium system composite material, any of a titanium dioxide, low hypo---ic acid-ized titanium,

and oxidization titanium nitride are sufficient. Moreover, to mica titanium system composite material or bismuth oxy-chloride, ferrous oxide, Berlin blue, chromic oxide, carbon black, carmine, or ultramarine blue may be mixed further.

[0032] As a metal, aluminum, iron, NIKKERRU, cobalt, chromium, gold, silver, copper, platinum, zinc, an indium, tin, antimony, a tungsten, a zirconium, molybdenum, silicon, titanium, etc. are mentioned, for example.

[0033] As magnetic fine particles, for example Gamma-Fe 2O3 and magnetite (Fe 3O4), Belt light system ferrous oxide (FeOx;1.33<x<1.5) or they Cobalt, What denaturalized with manganese, nickel, zinc, chromium, etc., the iron powder which iron, or aluminum, B, Co, Cr, Cu, Mo, Mn, nickel, P, Si, needlelike Sn and needlelike Zn contained, and CrO2 Ba ferrite etc. is mentioned.

[0034] Moreover, although powder may be the iron covered on the mica, nickel, cobalt, or its oxide, it is not limited to this.

[0035] as a silicate mineral -- a phyllosilicate mineral (for example, a kaolin group --) They are a montmorillonite group, a clay mica group, a chlorite group, serpentine, and a theque TOKEI acid chloride mineral (for example, zeolite group). Pyrophyllite, talc, chlorite, a chrysotile, antigorite, RIZADAITO, a kaolinite, dickite, nacrite, halloysite, A montmorillonite, nontronite, saponite, a sauconite, and a bentonite, Zeolites, such as heulandite groups, such as natrolite groups, such as natrolite, a mesolite, a SUKORESU zeolite, and a thomsonite, a heulandite, a stilbite, and ******, and analcime, a cross stone, an ashes cross-joint zeolite, chabazite, and a gmelinite, etc. are mentioned.

[0036] Although silicone covering of the porous matter can furthermore be performed good by this invention, after corning or casting the thing which corned or cast a porous glass bead, a hollow silica, a zeolite or a metallic oxide, a metal nitride, the silicate mineral, the carbonate mineral, the sulfate mineral, or the phosphate mineral, or the above-mentioned mineral as porous matter, for example, what was calcinated, metal, a cellulose, fiber, or synthetic resin can be mentioned.

[0037] Especially the approach of contact of fine particles and a mixed solution is not limited, and it can perform it by the approach of arbitration that fine particles are immersed into a mixed solution, for example, carry out the spray coating cloth of the mixed solution to fine particles.

[0038] In addition, the amount of silicone compounds to fine particles is 0.5 - 15 % of the weight preferably 0.1 to 20% of the weight. At a rate mentioned above to this amount of silicone compounds, a drainage system solution is mixed and it uses as reaction mixture (mixed solution).

[0039] Reaction mixture is preferably heated after the above-mentioned contact. Although it is desirable to also perform baking of a silicone coat to coincidence although especially heating conditions are not limited and heating at the temperature of about 70-120 degrees C may generally be enough for it, a bridge formation organic-functions rate increases above 95 degrees C. In addition, depending on the fine particles to be used, you may heat to about 400 degrees C. Since moisture can be evaporated and the crosslinking reaction of Si-H radicals can be promoted with this heating, stabilization of silicone covering can be attained.

[0040] In addition, when using what has a Si-H radical as a silicone compound in this invention, alkali can be added to the system of reaction and it can be made to promote bridge formation of this Si-H radical. As alkali to be used, alkaline earth metal, such as alkali metal, such as Li, Na, K, Rb, and Cs, and Mg, calcium, other organic amines, etc. are mentioned. Although the addition of these alkali metal, alkaline earth metal, etc. changes with rates of bridge formation of the Si-H radical for which it asks etc., generally, its about 0.01 - 5.0 % of the weight is desirable to fine particles, and it is 0.1 - 1.0 % of the weight more preferably. These may use one sort or may use it combining two or more sorts.

[0041] Moreover, pH adjustment of a system can be freely carried out by carrying out suitable amount addition of the acid after the above-mentioned crosslinking reaction, and neutralizing alkali. As an acid to be used, a fatty acid, the fatty acid which has a perfluoro radical are mentioned as a suitable thing. The degree of bridge formation of a Si-H radical is freely controllable by adjusting pH of a system. That is, if a system is leaned to an alkali side, bridge formation of a Si-H radical can be promoted, and on the other hand, if a system is leaned to an acid side, the degree of bridge formation can be stopped. Even when alkali is added, bridge formation of Si-H radicals is promoted and an acid neutralizes after that, if

the salt to generate is chosen, it is not necessary to filter, and is an economical art. Moreover, the covering fine particles which made pH the acid side, made bridge formation hard to produce and made [many] the Si-H radical of survival can raise the addition consistency, when performing a hydrosilylation reaction after that, introducing a pendant radical and giving functionality.
[0042] In addition, in the combination of alkali and an acid, if the complex to generate is water repellence, since the water repellence of silicone covering fine particles is not affected, it is desirable. As such a combination, myristic-acid calcium, calcium stearate, etc. are mentioned, for example.
[0043] Although addition of this alkali and an acid may be performed in any before heating and after heating, it is good to carry out after heating preferably.
[0044] As an example of the manufacture approach of this invention, after mixing the silicone compound and drainage system solution of the specified quantity with a mixer, fine particles are sprayed in the shape of a fog, and the approach of carrying out heating mixing is mentioned, for example. Moreover, after putting a drainage system solution into the wet-blending machine containing fine particles and mixing, a silicone compound may be added, heating mixing may be carried out, or silicone / drainage system solution may be directly added to fine particles, and heating mixing may be carried out.
[0045] In order according to the manufacture approach of this invention to use a drainage system solution instead of an organic solvent and to perform covering processing of fine particles, activity safety is high and does not need removal of the organic solvent which remains, either. moreover, there is also no problem of ignition, processing near a dry type condition is also markedly boiled compared with an organic solvent system, and safety is high. Moreover, when the silicone compound which has a Si-H radical is used, crosslinking reaction can fully be advanced by supplying a water molecule required for bridge formation of a Si-H radical.
[0046] Furthermore, a silicone coat can be covered with heating on a fine-particles front face to homogeneity and a stabilization target by burning silicone to coincidence.
[0047] Moreover, since covering processing can be carried out by the drainage system, without adding a surfactant etc., it is not necessary to wash and there is no fault at the time of using a surfactant.
[0048] Thus, the amount of unreacted Si-H base exists, and the made silicone covering fine particles become instability a little on severe conditions like alkali or an acid, when the silicone compound which has for example, a Si-H radical is used.
[0049] Therefore, in practical use, this Si-H radical and the compound which can react are added to the unreacted Si-H radical of the silicone compound which covered the above-mentioned fine particles, for example. Thereby, a Si-H reactivity compound is added to a part for the unreacted Si-H base of a silicone compound, and the pendant radical guided from a Si-H reactivity compound is introduced into a silicone compound. Various functions can be given to fine particles by choosing a Si-H reactivity compound appropriately and introducing a desired pendant radical.
[0050] Although bridge formation of Si-H radicals will arise on a fine-particles front face, the network structure will be formed in the above-mentioned covering process of the 1st step and a fine-particles front face will be covered with the coat of a silicone compound if this is described further, bridge formation is not completely performed for steric hindrance etc. Therefore, the Si-H radical of survival exists and there is an inclination which becomes unstable a little on severe conditions like alkali or an acid. Still more stable fine particles can be obtained to alkali or an acid by making Si-H reactivity compounds (for example, unsaturated compounds, such as an alkene and an alkyne etc.) add to the Si-H radical of this survival by the hydrosilylation reaction, and making Si-C association generate.
[0051] Therefore, various functions can be given to fine particles by choosing appropriately Si-H radical reactivity compounds (unsaturated compound etc.), and introducing a desired pendant radical. A "pendant radical" is the residue of a part for a Si-H base, and the compound which can react, and means the radical introduced into a silicone polymer by the addition reaction of the compound here. This pendant radical gives various kinds of properties and functions to fine particles. If a class or die length of a hydrocarbon group of an unsaturated compound made to add is adjusted, hydrophobicity can be strengthened more.

[0052] An example explains this invention further below at a detail. However, it cannot be overemphasized that the range of this invention is not what is limited in any way by these examples.
[0053]
[Example]
The example 1 sericite powder 200 weight section and the ion-exchange-water 200 weight section were put into the kneader, and for 10 minutes improved stirring mixing at the room temperature. Then, began the temperature up, continuing 6 weight ****** and stirring mixing for methil hydrogen polysiloxane (product made from "silicone KF-99" Shin-etsu Chemistry), it was made to react at 100 degrees C for 5 hours, and water was fully removed. In this way, the made silicone covering mica showed remarkable hydrophobicity.
[0054] It put into the kneader who kept warm the example 2 sericite powder 200 weight section, the calcium-hydroxide 0.5 weight section, and the ion-exchange-water 150 weight section at 70 degrees C, and for 10 minutes improved stirring mixing. Then, after carrying out 3 weight sections addition of the methil-hydrogen-polysiloxane (product made from "silicone KF-99" Shin-etsu Chemistry) 3 weight section, and the phenylmethyl polysiloxane (product made from "silicone KF-56" Shin-etsu Chemistry) and carrying out a temperature up to 100 degrees C, it stirred for 1 hour, and the palmitic-acid 3 weight section was added, and stirring was continued for further 5 hours. In this way, the made silicone covering sericite showed remarkable hydrophobicity.
[0055] The example 3 sericite powder 100 weight section, the talc powder 70 weight section, the red ferrous-oxide 2 weight section, the Synthetic Ochre 6 weight section, the black-iron-oxide 0.2 weight section, the titanium-dioxide 10 weight section, and the ion-exchange-water 150 weight section were put into the kneader, and for 10 minutes improved stirring mixing at the room temperature. Then, the methil-hydrogen-polysiloxane (product made from "silicone KF-99" Shin-etsu Chemistry) 6 weight section was added, the temperature up was begun, continuing stirring mixing, and it was made to react at 100 degrees C for 5 hours. In this way, the made silicone covering composite powder object showed remarkable hydrophobicity.
[0056] The example 4 sericite powder 100 weight section, the talc powder 70 weight section, the red ferrous-oxide 2 weight section, the Synthetic Ochre 6 weight section, the black-iron-oxide 0.2 weight section, the titanium-dioxide 10 weight section, the magnesium-hydroxide 0.2 weight section, and the ion-exchange-water 150 weight section were put into the kneader, and for 10 minutes improved stirring mixing at the room temperature. Then, the methil-hydrogen-polysiloxane (product made from "silicone KF-99" Shin-etsu Chemistry) 8 weight section was added, after carrying out a temperature up to 100 degrees C, it stirred for 1 hour, and the stearin acid 3 weight section was added, and stirring was continued for further 5 hours. In this way, the made silicone covering composite powder object showed remarkable hydrophobicity.
[0057] The example 5 sericite powder 100 weight section, the titanium-dioxide 130 weight section, the kaolin 250 weight section, the talc 350 weight section, the red iron-oxide 10 weight section, the Synthetic Ochre 25 weight section, the black-iron-oxide 1 weight section, and the ion-exchange-water 700 weight section were put into the kneader, and for 10 minutes improved stirring mixing at the room temperature. Then, the methil-hydrogen-polysiloxane (product made from "silicone KF-99" Shin-etsu Chemistry) 30 weight section was added, after carrying out a temperature up to 100 degrees C, it stirred for 3 hours, and reduced pressure drying was performed after that for 3 hours. In this way, the made silicone covering composite powder object showed remarkable hydrophobicity.
[0058] The example 6 titanium-dioxide 100 weight section, the calcium-hydroxide 0.3 weight section, and the ion-exchange-water 80 weight section were put into the kneader, and for 10 minutes improved stirring mixing at the room temperature. Then, the methil-hydrogen-polysiloxane (product made from "silicone KF-99" Shin-etsu Chemistry) 3 weight section was added, after carrying out a temperature up to 100 degrees C, it stirred for 1 hour, and 3 weight sections, in addition 2-hour stirring were performed for the myristic acid, and reduced pressure drying was performed after that for 3 hours. In this way, the made silicone covering titanium dioxide showed remarkable hydrophobicity.
[0059] The silicone covering composite powder object fine-particles 86.3 weight section of example 7

example 5 and the glycerol 2.0 weight section were put into the high-speed blender, and it mixed. Apart from this, the liquid paraffin 8 weight section, the sorbitan sesquioleate 3.5 weight section, and the ethylparaben 0.2 weight section were mixed, and what carried out heating homogeneity was added to the above-mentioned mixture, and it mixed to homogeneity further. This was processed with the grinder, the screen was pressed after preparing through grain size, and cake mold foundation was obtained. The obtained foundation had good makeup ****.

[0060]

Example 8 : Emulsification foundation A combination component Weight % (A) ion exchange water 43.5 Sodium chondroitin sulfate 1.0 Sodium lactate 0.5 1, 3-butylene glycol 3.0 Methylparaben ** Amount (B) dimethylpolysiloxane (20cs) 16.0 Decamethyl cyclopentasiloxane 5.0 Silicone resin 1.0 cetyl iso OKUTANETO 1.0 polyoxyalkylene denaturation organopolysiloxane 4.0 (20% of rates of denaturation)

An antioxidant ** Amount Perfume ** Covering fine particles of the amount (C) example 2 15.0 Covering fine particles of an example 4 Addition distribution of the fine particles of a component (C) was carried out for 10.0 process components (B) after the heating dissolution. Addition emulsification of the component (A) which carried out the melt heat further beforehand was carried out, it cooled to the room temperature, and emulsification foundation was obtained.

[0061] In the formula of example of comparison 1 example 8, the emulsification foundation of the example 1 of a comparison was obtained like the example 8 except having replaced the silicone covering fine particles of the examples 2 and 4 in a component (C) with in the end of unsettled composite powder.

[0062] Compared with the thing of the example 1 of a comparison, the dispersibility of a colored pigment was good, the thing of an example 8 was finished finely and its sunscreen effectiveness was high.

[0063]

Example 9 : Presto powder A combination component The covering fine particles of the weight % (1) example 2 The covering fine particles of the 90.0 (2) example 4 5.9 (3) squalane 2.0(4)2-ethylhexyl palmitate 2.0 (5) perfume The 0.1 process above-mentioned component (1) and (2) were mixed with the Henschel mixer, what carried out heating mixing of a component (3) and (4) was sprayed on this, and it ground after mixing, it cast to the inside pan, and presto powder was obtained. The obtained presto powder had good makeup ****.

[0064]

Example 10 : An ultraviolet-rays defense stick A combination component The covering fine particles of the weight % (1) example 6 The covering fine particles of the 15.0 (2) example 2 The covering fine particles of the 20.0 (3) example 4 6.5 (4) carnauba waxes 1.0 (5) solid paraffin 3.0 (6) liquid paraffins 45.0 (7) isopropyl myristate 8.0 (8) sorbitansesquiolate 1.5 (9) perfume ** The amount process above-mentioned component (6), (7), and (8) are slushed into an iron pot, and it warms at 80-90 degrees C, and a component (4) and (5) were added and it was made to dissolve. Component (1) - (3) was added to this, and it distributed to homogeneity, and after degassing, the component (9) was added and it stirred gently. The ultraviolet-rays defense stick was obtained by slushing this into a container at 80 degrees C, and cooling to a room temperature. The ultraviolet-rays defense effectiveness of the obtained stick was high.

[0065]

[Effect of the Invention] Since fine particles are contacted to the mixed solution which mixed the silicone compound to the drainage system solvent in the manufacture approach of silicone covering fine particles in this invention and this was further heated by request as explained in full detail above While being able to attain reduction-ization of a manufacturing cost, the effectiveness which mitigation-ized the burden also in environmental sides, such as wastewater, that the manufacture approach of safe and stable silicone covering fine particles can be offered is done so. Moreover, improvement in the manufacture effectiveness of the product using the covering fine particles obtained by the above-mentioned manufacture approach, reduction of a manufacturing cost, reservation of the stability of

product quality and safety, mitigation of the load on an activity in the economy top list in respect of an environmental cure, etc. can be aimed at.

[Translation done.]